
लौह प्रबलित आयोडीनकृत नमक —
खाद्य ग्रेड — विशिष्टि

Iron Fortified Iodized Salt (Double
Fortified Salt) — Food Grade —
Specification

ICS 67.220.20

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Food Additives Sectional Committee had been approved by the Food and Agriculture Division Council.

The diet of more than a billion people in the developing countries is deficient in two key micro nutrients: iodine and iron. Low iodine levels in the diet lead to iodine deficiency disorders, while low iron intake leads to anemia, which reduces work capacity and resistance to disease. Iron deficiency anemia is a major nutritional problem in our country. The vulnerable groups most affected are women of reproductive age, pregnant women and pre-school children. The major cause of anemia in our country is iron deficiency due to inadequate intake of dietary iron and/or its poor absorption from predominantly cereal based diets consumed by our population.

Deficiencies in iodine or iron can be inexpensively eliminated, prevented, or reduced by increasing their dietary intake through fortification of food. However, it is critical that the carrier for the micronutrients be available to the entire population, and it must be consumed at a constant rate independently of social or economic status of each consumer.

Salt is an attractive vehicle in the provision of both iodine and iron, because of its uniform consumption and availability to all segments of the population. In India, edible common salt has been identified as a suitable material for fortification by iron as well as iodine as it is consumed in fairly uniform quantity by every section of the population. Given the almost universal use of salt as a condiment, double fortified salt has the potential to alleviate iodine and iron deficiencies in billions of people. This standard has been formulated to cover the requirements for iron fortified iodized salt (Double fortified salt), food grade.

In the formulation of this standard, due consideration has been given to *the Food Safety and Standards Act, 2006* and Regulations framed thereunder and *Legal Metrology (Packaged Commodities) Rules, 2011*. The standard is however subject to restrictions imposed under these Rules, wherever applicable.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

IRON FORTIFIED IODIZED SALT (DOUBLE FORTIFIED SALT) — FOOD GRADE — SPECIFICATION

1 SCOPE

This standard prescribes requirements, methods of sampling and test for iron fortified iodized salt (Double fortified salt), food grade.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
253 : 2014	Specification for common salt (<i>fourth revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
2711 : 1979	Direct reading pH meters (<i>second revision</i>)
4905 : 1968	Methods for random sampling

<i>IS No.</i>	<i>Title</i>
7224 : 2006	Iodized salt, vacuum evaporated iodized salt and refined iodized salt — Specification (<i>second revision</i>)

3 REQUIREMENTS

3.1 Description

Iron fortified iodized salt (Double fortified salt) shall be crushed crystalline solid; white or pale pink or light grey in colour, free from contamination with clay and other extraneous adulterants and impurities.

3.2 Salt used for manufacture of double fortified salt shall have minimum 98.0 percent of sodium chloride content on dry weight basis and moisture not more than 1.5 percent when tested in accordance with the method prescribed in **A-5** of IS 253 and Annex A of IS 7224, respectively.

3.3 Ninety five percent of the material shall pass through 4.00 mm IS sieve.

3.4 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Iron Fortified Iodized Salt (Double Fortified Salt)
(Clause 3.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex (4)	IS No. (5)
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, <i>Max</i>	1.5	—	Annex A of IS 7224
ii)	Water insoluble matter, percent by mass on dry basis, <i>Max</i>	1.0	—	A-4 of IS 253
iii)	Chloride content (as NaCl), percent by mass, on dry basis, <i>Min</i>	97.0	—	A-5 of IS 253
iv)	Matter insoluble in dilute HCl, percent by mass on dry basis, <i>Max</i>	0.30	—	A-6 of IS 253
v)	Matter soluble in water other than sodium chloride, percent by mass, on dry basis, <i>Max</i>	2.5	—	A-7 of IS 253
vi)	Iron content (as Fe), mg/kg	850 - 1 100	A	—
vii)	Iodine content, mg/kg, <i>Min</i> : a) Manufacturer's level	30	B	—
		15	B	—
viii)	pH	3.5 - 5.5	C	—
ix)	Sulphate (as SO ₄), percent by mass, <i>Max</i>	1.1	—	A-9 of IS 253
x)	Magnesium (as Mg), water soluble, percent by mass, <i>Max</i>	0.10	—	A-8 of IS 253
xi)	Phosphorous (as P ₂ O ₅), mg/kg	2 800 - 3 100	D	—
xii)	Sodium hexa meta phosphate, percent by mass on dry basis, <i>Max</i>	1.0	E	—

4 PACKING

4.1 The material shall be packed in HDPE bags or any other suitable packages, as agreed to between the purchaser and the supplier.

4.2 For bulk packings, the material may be packed in 40, 50, 75 and 100 kg packages.

4.3 For small packings the material may be packed in 50g, 100g, 200g, 500g, 1 kg, 2 kg and 5 kg suitable packages. For consumer packing, the material shall be packed in food grade plastics. The thickness of the plastic film used for packing shall be not less than 50µ.

5 MARKING

5.1 The packages shall be securely closed and legibly and clearly marked with the following particulars:

- a) Name 'IRON FORTIFIED IODIZED SALT (DOUBLE FORTIFIED SALT)';
- b) Name and address of the manufacture;
- c) Batch or Code number;
- d) Iron content;
- e) Iodine content when packed;
- f) Date of manufacturing (month and year);
- g) Best before date;
- h) Net quantity;
- j) Instructions for storage 'Store in a cool and

dry place'; and

- k) Any other details required under the under the *Legal Metrology (Packaged commodities) Rules, 2011* and *Food Safety & Standards (Packaging and Labelling) Regulation, 2011*.

5.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

5.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative samples of the material for testing its conformity to this standard shall be drawn according to the method prescribed in Annex F.

7 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, Sl No. (vi)]

DETERMINATION OF IRON IN DOUBLE FORTIFIED SALT

A-0 PRINCIPLE

Iron is determined colorimetrically by the principle that ferric ion (Fe^{3+}) gives a blood red colour with potassium thiocyanate (KCNS).

A-1 APPARATUS

A-1.1 Spectrophotometer

A-2 REAGENTS

A-2.1 Sulphuric Acid (30 Percent) — Take 60 ml of distilled water in a beaker. Keep in an ice bath and add slowly drop-wise 30 ml of concentrated H_2SO_4 with constant stirring. Make up the volume to 100 ml with distilled water.

A-2.2 Potassium Persulphate (7 Percent) — Dissolve 7g of $\text{K}_2\text{S}_2\text{O}_8$ in distilled water and make up the volume to 100 ml with distilled water.

A-2.3 Potassium Thiocyanate (40 Percent) — Dissolve 40g of KCNS in 90 ml distilled water. Add 4 ml acetone and make up the volume to 100 ml.

A-2.4 Standard Iron Solution — Dissolve 702.2 mg ferrous ammonium sulphate in 100 ml distilled water. Add 5 ml of 1:1 hydrochloric acid (HCl) and make up the volume to 1 000 ml (0.1 mg iron/ml). The standard solution is prepared fresh once in 6 months. From this prepare working standard.

A-2.5 Working standard (10 μg Iron/ml) — Dilute

10 ml of the standard iron solution (0.1 mg iron/ml) to 100 ml with distilled water. This will give 10 μg iron/ml concentration.

A-3 PROCEDURE

A-3.1 Take 1 g of double fortified salt in a 100 ml standard measuring flask using a glass funnel. Add 2.5 ml of concentrated HCl and make up the volume to 100 ml with distilled water. Mix and use 1 ml or 2 ml aliquots for the estimation of iron as given under Table 2.

A-3.2 Standard Calibration Graph

Measure the red colour developed within 20 min at 540 nm. Add 40 percent KCNS solution just before taking the readings. Draw a standard graph of the iron standards by taking iron concentration (μg iron) on the X-axis and the OD on the Y-axis and calculate the iron content from the standard graph (see Fig. 1).

A-4 CALCULATION

$$\text{Iron (as Fe), mg/kg} = \frac{M_1 \times 100}{M}$$

where

M_1 = mass of iron (as Fe) in 100 ml of the sample solution, in mg; and

M = mass of the material taken for the test, in g.

Table 2 Preparation of Samples
(Clause 3.1)

Sl No.	Reagent	Test Tube 1	Test Tube 2	Test Tube 3	Test Tube 4	Test Tube 5	Test Tube 6	Test Tube 7	Test Tube 8
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
i)	Distilled water, ml	6.5	5.5	4.5	6.0	5.5	4.5	3.5	2.5
ii)	Iron working standard, ml	0	0	0	0.5	1.0	2.0	3.0	4.0
iii)	DFS solution, ml	0	1.0	2.0	0	0	0	0	0
iv)	30 percent H_2SO_4 , ml	1	1	1	1	1	1	1	1
v)	7 percent ($\text{K}_2\text{S}_2\text{O}_8$), ml	1	1	1	1	1	1	1	1
vi)	40 percent KCNS, ml	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
vii)	Total volume, ml	10	10	10	10	10	10	10	10

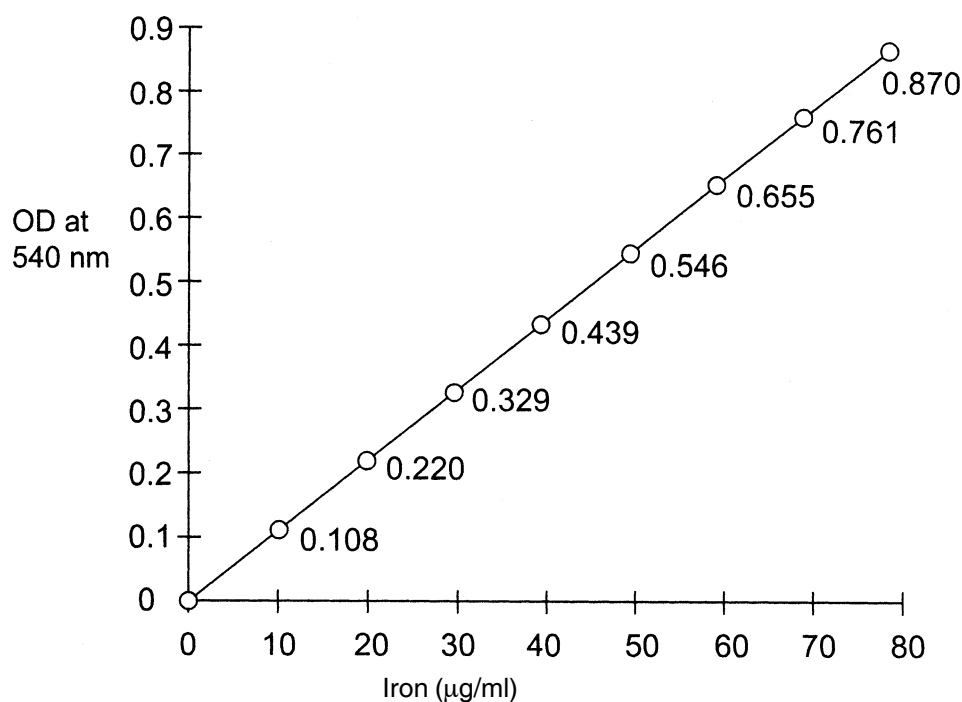


FIG. 1 STANDARD GRAPH FOR IRON ESTIMATION

ANNEX B

[Table 1, Sl No. (vii)]

DETERMINATION OF IODINE IN DOUBLE FORTIFIED SALT

B-0 PRINCIPLE

The iodine content of DFS is measured by a modified iodometric titration using orthophosphoric acid.

B-0.1 Liberation of Free Iodine from Salt

Addition of ortho phosphoric acid liberates free iodine from iodate in DFS. Excess potassium iodide (KI) is added to help solubilize the free iodine, which is quite insoluble in pure water under normal conditions.

B-0.2 Titration of Free Iodine with Thiosulphate

Free iodine is consumed by sodium thiosulphate in the titration step. The amount of thiosulphate consumed is proportional to the amount of free iodine liberated from the DFS. Starch is added as external indicator of the reaction, and reacts with free iodine to produce a blue colour. When added towards the end point of titration (that is, when only a trace amount of free iodine

is left) the disappearance of blue colour, or end point, indicates that all remaining free iodine has been consumed by thiosulphate.

B-1 REAGENTS

B-1.1 Water— Use double distilled water or de-ionized water for the preparation of reagents for this method.

B-1.2 Potassium Iodide (1 percent) — Dissolve 1 g potassium iodide (KI) (Laboratory grade) in 100 ml water. Store in a cool, dark place. This volume is sufficient for 200 samples. The solution is stable at least for 3 months, if stored properly.

B-1.3 Ortho Phosphoric Acid (H_3PO_4) — Slowly add 75.4 ml of analytical grade ortho phosphoric acid (Sp. Gr. 1.75) to 900 ml of ice-cold distilled water. Dilute and make to 1 000 ml, with water. This volume is sufficient for 200 samples. The solution is stable indefinitely.

NOTE — Always add acid to water drop-wise, not water to acid. Stir solution while adding acid.

B-1.4 0.005M Sodium Thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) — Dissolve 1.24g $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ (Analytical grade) in 1 000 ml water. Store in a cool, dark place. This volume is sufficient for nearly 200 samples. The solution is stable at least for 1 month, if stored properly.

B-1.5 Starch Indicator Solution

B-1.5.1 Preparation of Saturated NaCl Solution — Make 100 ml of a saturated NaCl solution, by adding analytical grade NaCl in small quantities at a time, to approximately 80 ml water in a beaker, with stirring and heating, until no further NaCl will dissolve. This solution is stable for at least one year.

B-1.5.2 Preparation of Starch — Weigh 1 g soluble starch (Potato, extrapure/laboratory grade) into a 100 ml beaker, add 10 ml water and make a paste, heat to dissolve. Add saturated NaCl solution to the hot starch solution to make up to 100 ml. Store in a cool/dark place. This volume is sufficient for 200 samples. The solution is stable for up to one month, and should be heated (not boiled) each day before use to re-suspend any solids (NaCl).

B-1.6 Standard Potassium Iodate (KIO_3) Solution — Weigh accurately 0.167 g of KIO_3 (analytical grade) and dissolve in water in a standard

measuring flask (100 ml) and make up the volume to 100 ml. This will give a concentration of 1 mg of iodine/ml.

B-2 PROCEDURE

B-2.1 Double Fortified Salt Sample

Run salt samples as well as standards in duplicates.

B-2.1.1 Weigh accurately 10 g of double fortified salt into a 250 ml Erlenmeyer flask with a stopper. Add 0.5 ml 1 percent KI solution. Add 46 ml water. Swirl the flask to dissolve the salt. Add 5 ml of 4N H_3PO_4 . The solution will turn yellow, if iodine is present. Stopper the flask and put in the dark (cup-board) for 10 min.

B-2.1.2 Rinse and fill the burette with 0.005M $\text{Na}_2\text{S}_2\text{O}_3$ and adjust level to zero. Remove flask from the dark and titrate against $\text{Na}_2\text{S}_2\text{O}_3$ from the burette until the solution turns pale yellow (straw yellow). Add 0.5 ml of starch indicator solution (the solution shall turn dark blue) and continue titration until the solution becomes colourless. Record the volume of thiosulphate in the burette and convert to mg/kg using the Table 3.

NOTE — Do not pipette by mouth. The reaction mixture should be kept in the dark before titration because a side reaction can occur when the solution is exposed to light that causes iodide ions to be oxidized to iodine.

Table 3 Conversion Table for Iodine Content

(Clause B-2.2)

Sl No.	Burette Reading ml	Iodine mg/kg	Burette Reading ml	Iodine mg/kg	Burette Reading ml	Iodine mg/kg	Burette Reading ml	Iodine mg/kg
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
i)	0.0	0.0	2.5	26.5	5	52.9	7.5	79.4
ii)	0.1	1.1	2.6	27.5	5.1	54.0	7.6	80.4
iii)	0.2	2.1	2.7	28.6	5.2	55.0	7.7	81.5
iv)	0.3	3.2	2.8	29.6	5.3	56.1	7.8	82.5
v)	0.4	4.2	2.9	30.7	5.4	57.1	7.9	83.6
vi)	0.5	5.3	3.0	31.7	5.5	58.2	8.0	84.6
vii)	0.6	6.3	3.1	32.8	5.6	59.2	8.1	85.7
viii)	0.7	7.4	3.2	33.9	5.7	60.3	8.2	86.8
ix)	0.8	8.5	3.3	34.9	5.8	61.4	8.3	87.8
x)	0.9	9.5	3.4	36.0	5.9	62.4	8.4	88.9
xi)	1.0	10.6	3.5	37.0	6.0	63.5	8.5	89.9
xii)	1.1	11.6	3.6	38.1	6.1	64.5	8.6	91.0
xiii)	1.2	12.7	3.7	39.1	6.2	65.6	8.7	92.0
xiv)	1.3	13.8	3.8	40.2	6.3	66.7	8.8	93.1
xv)	1.4	14.8	3.9	41.3	6.4	67.7	8.9	94.2
xvi)	1.5	15.9	4.0	42.3	6.5	68.8	9.0	95.2
xvii)	1.6	16.9	4.1	43.4	6.6	69.8	9.1	96.3
xviii)	1.7	18.0	4.2	44.4	6.7	70.9	9.2	97.3
xix)	1.8	19.0	4.3	45.5	6.8	71.9	9.3	98.4
xx)	1.9	20.1	4.4	46.6	6.9	73.0	9.4	99.5
xxi)	2.0	21.2	4.5	47.6	7.0	74.1	9.5	100.5
xxii)	2.1	22.2	4.6	48.7	7.1	75.1	9.6	101.6
xxiii)	2.2	23.3	4.7	49.7	7.2	76.2	9.7	102.6
xxiv)	2.3	24.3	4.8	50.8	7.3	77.2	9.8	103.7
xxv)	2.4	25.4	4.9	51.9	7.4	78.3	9.9	104.7

B-2.2 Standardization of Potassium Iodate (KIO_3) Solution

Run standard KIO_3 (1 mg iodine/ml) with 10 g of non-iodized plain salt as a part of control. Take 46 ml water into a 250 ml Erlenmeyer flask with a stopper. Add 1 ml of standard KIO_3 (1 mg iodine/ml) and 10 g non-iodized plain salt. Mix well to dissolve the salt. Add 0.5 ml 1 percent KI. Add 5 ml 4N H_3PO_4 . Stopper the flask and put in the dark for 10 min. Then titrate against 0.005M $\text{Na}_2\text{S}_2\text{O}_3$ (see B-2.1.2) and calculate the iodine

content using the Table 3. This shall give an iodine value of 100 ppm (1 000 mg/10g) (see Fig. 2).

NOTES

1 Inaccurate results may occur, if starch solution is used while still warm.

2 If starch indicator is added too early, a strong iodine-starch complex is formed, which reacts slowly, and gives falsely elevated results.

3 The reaction should be performed at room temperature ($<30^\circ\text{C}$), since the iodine is volatile, and the indicator solution loses sensitivity when exposed to high temperature.

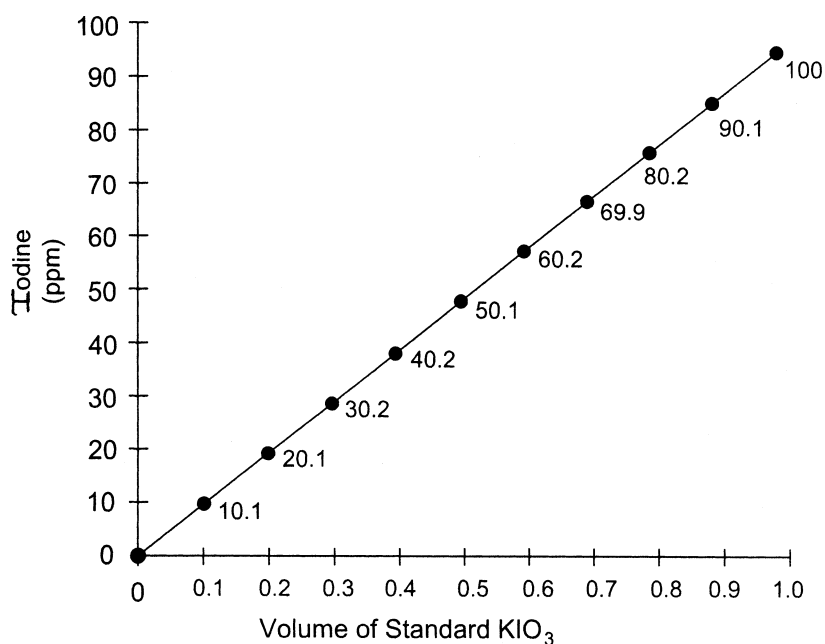


FIG. 2 POTASSIUM IODATE (KIO_3) STANDARD GRAPH

ANNEX C

[Table 1, Sl No. (viii)]

DETERMINATION OF pH

C-1 APPARATUS

C-1.1 pH Meter — Use a standard laboratory pH meter (see IS 2711).

C-2 PROCEDURE

Dissolve 5 g of the sample in 100 ml water. Measure the pH of the solution using a standard pH meter. Repeat the test and record the mean pH value.

ANNEX D

[Table 1, Sl No. (xi)]

ESTIMATION OF PHOSPHOROUS (P_2O_5) CONTENT IN IRON FORTIFIED IODIZED
(DOUBLE FORTIFIED) SALT

D-1 REAGENTS

D-1.1 Ammonium Molybdate (10 Percent w/v) — Dissolve 10g of ammonium molybdate $[(NH_4)_6Mo_7O_{24} \cdot 4H_2O]$ in 100 ml distilled water (DW).

D-1.2 Dilute Nitric Acid (≈ 2 N) — Mix 190 ml of 50 percent Nitric acid with distilled water to produce 1 000 ml.

D-1.3 Nitric Acid (2 Percent) — Dilute 10 ml of concentrated nitric acid (70 percent) to 710 ml with distilled water.

D-1.4 Potassium Nitrate (1 Percent) — Dissolve 1 g potassium nitrate in 100 ml of distilled water.

D-1.5 Phenolphthalein — Dissolve 0.2 g of phenolphthalein ($C_{20}H_{14}O_4$) in 60 ml of 90 percent ethanol and add sufficient distilled water to produce 100 ml.

D-1.6 1N Sodium Hydroxide — Dissolve 4 g sodium hydroxide in 100 ml distilled water. Prepare fresh daily for use.

D-1.7 1N Sulphuric Acid — Dilute 3 ml of concentrated sulphuric acid to 100 ml in distilled water.

D-2 PROCEDURE

Dissolve 10g of double fortified salt in 50 ml distilled water in a 500 ml beaker. Add 5ml of concentrated nitric acid and stir well. Add 220 ml of distilled water kept at 60°C. Stir well. Wait for 10 min. Add 10g of ammonium nitrate (purified). At 50°C, add 100 ml of 10 percent ammonium molybdate (freshly prepared) and stir well with a glass rod for 10 min. Repeat stirring after 1minute gap. Keep for 30 min.

Filter in a weighed Gooch Crucible (G4) using 2 percent nitric acid. Make sure that the entire precipitate is transferred to the crucible. Wash 3-4 times the precipitate in the crucible with 2 percent nitric acid. Find the weight of the precipitate and calculate the P_2O_5 content.

Alternatively, filter the precipitate using Whatman No. 40 circles and wash with 10 ml of dilute nitric acid (≈ 2 N), followed by potassium nitrate solution (1 percent) until the filtrate is no longer acidic to litmus (Blue litmus remains blue). Dissolve the precipitate from the filter paper in 60 ml of 1N sodium hydroxide, add phenolphthalein, and titrate the excess sodium hydroxide with 1N sulphuric acid. Calculate the volume of 1N sodium hydroxide consumed by the precipitate. Each ml of 1N sodium hydroxide consumed is equivalent to 0.003 086 g P_2O_5 .

D-3 CALCULATION

Phosphorous as P_2O_5 , mg/kg = $p \times 0.044$

where

p = phosphorous as P_2O_5 percent by mass (on dry basis) to be calculated as follows:

$$\text{Phosphorous as } P_2O_5 \text{ percent by mass (on dry basis) } N_2 = p = 60 - \left(\frac{V_1 \times N_1}{N_2} \right) \times 3.1$$

where

V_1 = volume of H_2SO_4 (titre value),

N_1 = normality of H_2SO_4 , and

N_2 = normality of NaOH.

ANNEX E

[Table 1, Sl No. (xii)]

METHOD OF ASSAY OF SODIUM HEXA META PHOSPHATE

E-1 APPARATUS

Standard volumetric flask, Pipette, beaker, Gooch Crucible, Whatman No. 40 filter paper/circles.

E-2 PREPARATION OF SAMPLE

Dry about 10 g of sodium hexa meta phosphate at 110°C overnight. Keep in a dessicator and bring to room temperature. Take 1 g (accurately weighed) in a 100 ml beaker.

E-3 REAGENTS

E-3.1 Ammonium Molybdate (10 Percent w/v) — Dissolve 10 g of ammonium molybdate $[(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}]$ in 100 ml distilled water.

E-3.2 Dilute Nitric Acid (≈ 2 N) — Mix 190 ml of 50 percent nitric acid with distilled water to produce 1 000 ml.

E-3.3 Nitric Acid (2 Percent) — Dilute 10 ml of concentrated nitric acid (70 percent) to 710 ml with distilled water.

E-3.4 Potassium Nitrate (1 Percent) — Dissolve 1 g potassium nitrate in 100 ml of distilled water.

E-3.5 Phenolphthalein — Dissolve 0.2 g of phenolphthalein ($\text{C}_{20}\text{H}_{14}\text{O}_4$) in 60 ml of 90 percent ethanol and add sufficient distilled water to produce 100 ml.

E-3.6 Sodium hydroxide (1N) — Dissolve 4 g sodium hydroxide in 100 ml distilled water. Prepare fresh daily for use.

E-3.7 Sulphuric Acid (1N) — Dilute 3 ml of concentrated sulphuric acid to 100 ml in distilled water.

E-4 PROCEDURE

E-4.1 Dry about 10 g of sodium hexa meta phosphate at 110°C overnight. Keep in a desiccator and bring to room temperature. Take 1 g (accurately weighed) in a 100 ml beaker. Dissolve in 25 ml of distilled water.

Transfer to a 500 ml standard volumetric flask. Make up the volume to 500 ml with distilled water (1g sodium hexa meta phosphate /500 ml). Pipette out exactly 25 ml (50 mg sodium hexa meta phosphate) into a 500 ml beaker. Add 5ml of concentrated nitric acid and stir well. Add 220 ml of distilled water kept at 60°C. Stir well. Wait for 10 min. Add 10g of ammonium nitrate (purified). At 50°C, add 100 ml of 10 percent ammonium molybdate (freshly prepared) and stir well with a glass rod for 10 min. Repeat stirring after 1 min gap. Keep for 30 min. Filter in a weighed Gooch Crucible (G4) using 2 percent nitric acid. Make sure that the entire precipitate is transferred to the crucible. Wash 3-4 times the precipitate in the crucible with 2 percent nitric acid. Find the weight of the precipitate and calculate the P_2O_5 content.

E-4.2 Alternatively, filter the precipitate using Whatman No. 40 circles and wash with 10 ml of dilute nitric acid ($\approx 2\text{N}$), followed by potassium nitrate solution (1 percent) until the filtrate is no longer acidic to litmus (Blue litmus remains blue). Dissolve the precipitate from the filter paper in 60 ml of 1N sodium hydroxide, add phenolphthalein, and titrate the excess sodium hydroxide with 1N sulphuric acid. Calculate the volume of 1N sodium hydroxide consumed by the precipitate. Each ml of 1N sodium hydroxide consumed is equivalent to 0.003 086 g P_2O_5 .

E-5 CALCULATION

$$\text{Sodium hexa meta phosphate as } \text{P}_2\text{O}_5 \text{ percent by mass on dry basis} = \left[60 - \left(\frac{V_1 \times N_1}{N_2} \right) \right] \times \left(\frac{0.06172}{W} \right) \times 100$$

where

V_1 = volume of H_2SO_4 (titre value);

N_1 = normality of H_2SO_4 ;

N_2 = normality of NaOH; and

W = mass of the material taken for the test, in g.

ANNEX F

(Clause 5.1)

SAMPLING OF IRON FORTIFIED IODIZED (DOUBLE FORTIFIED) SALT

F-1 GENERAL REQUIREMENTS OF SAMPLING

F-1.0 In drawing, storing, preparing and handling test samples, the following precautions shall be observed.

F-1.1 Samples shall not be taken out in an exposed place.

F-1.2 Precautions shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

F-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

F-1.4 The sample shall be placed in suitable, clean, dry and air-tight glass containers.

F-1.5 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling and year of manufacture.

F-2 SCALE OF SAMPLING

F-2.1 Lot

All the packages in a single consignment of iron fortified common salt drawn from a single batch of manufacture shall constitute a lot. If the consignment consists of different batches, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots.

F-2.1.1 The number of packages (n) to be selected shall depend upon the size of the lot (N) and shall be in accordance with Table 4.

F-2.1.2 These packages shall be selected at random from the lot, and to ensure randomness of selection, a random number table (*see* IS 4905) may be used.

F-3 PREPARATION OF TEST SAMPLES

F-3.1 Packages

F-3.1.1 From each of the packages selected accordance with **F-2.1.2**, a suitable portion of the material (about 200 g) shall be drawn with the help of a suitable sampling instrument.

F-3.1.2 Out of these portions, equal quantities of the material shall be taken and mixed thoroughly to form a composite sample of about 600 g. The composite samples shall be divided into three equal parts, namely,

Table 4 Number of Packages to be Selected for Sampling

(Clause F-2.1.1)

Sl No.	Lot Size n	No. of Packages to be Selected n
(1)	(2)	(3)
i)	4 to 50	3
ii)	51 to 100	4
iii)	101 to 150	5
iv)	151 to 300	7
v)	301 and above	10

NOTE — When the size of the lot is 3 or less, the number of packages to be selected and the criterion for judging conformity for the lot to the specification shall be as agreed to between the purchaser and the supplier.

one for the purchaser, the other for the supplier and the third to be used as a referee sample.

F 3.1.3 The remaining portion of the material from each container shall be divided into three equal parts, each forming an individual sample. One set of individual sample representing the container samples shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

F-3.2 All the individual samples and the composite sample shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

F-3.3 The referee test samples consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at place agreed to between the purchaser and the supplier, to be used in case of dispute.

F-4 NUMBER OF TESTS

F-4.1 Tests for the determination of moisture content, chloride content (as NaCl) and level of iron shall be performed on each of the individual samples.

F-4.2 Tests for the determination of all other characteristics given under **3** shall be performed on the composite sample (*see* **F-3.1.2**).

F-5 CRITERIA FOR CONFORMITY

F-5.1 For Individual Samples

From the test results the average ($\bar{x}$) and the range (R) or (R) shall be computed for each of the characteristics mentioned in **F-4.1**. Range (R) is defined as the

difference between the maximum and the minimum values of the individual test results. Range (R) is the average of two ranges calculated on each of the two sets of five consecutive test results when the total number of test results on a characteristic is 10. The criterion for conformity of the lot in respect of each of these characteristics is that $(\bar{x} - 0.6 R)$ or $(\bar{x} - 0.6 R)$ should be greater than or equal to the corresponding minimum requirements specified under **3**.

F-5.2 For Composite Samples

The lot shall be considered to have passed in respect of the characteristics tested on the composite test sample, if the test results satisfy the corresponding requirements given under **3**.

F-5.3 The lot shall be considered as conforming to the specification, if it satisfies all the criteria given in **F-5.1** and **F-5.2**.

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Amendments Issued Since Publication

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AMENDMENT NO. 1 AUGUST 2015
TO
IS 16232 : 2014 IRON FORTIFIED IODIZED SALT
(DOUBLE FORTIFIED SALT) — FOOD GRADE — SPECIFICATION

[Page 1, clause 3.2] — Substitute the following for the existing clause:

3.2 Salt used for manufacture of double fortified salt shall have minimum 98.0 percent of sodium chloride content on dry weight basis for double fortified salt manufactured by fortification with encapsulated ferrous fumarate and 99.0 percent of sodium chloride on dry weight basis for other forms of iron fortification when tested in accordance with the method prescribed in A-5 of IS 253. Moisture shall not be more than 1.5 percent by mass when tested in accordance with the method prescribed in Annex A of IS 7224 respectively.

[Page 1, Table 1] — Substitute the following for the existing table:

Table 1 Requirements for Iron Fortified Iodized Salt (Double Fortified Salt)
(Clause 3.4)

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, Max	1.5	—	Annex A of IS 7224
ii)	Water insoluble matter, percent by mass on dry basis, Max	1.0	—	A-4 of IS 253
iii)	Chloride content (as NaCl), percent by mass, on dry basis, Min	97.0	—	A-5 of IS 253
iv)	Matter insoluble in dilute HCl, percent by mass on dry basis, Max	0.30	—	A-6 of IS 253
v)	Matter soluble in water other than sodium chloride, percent by mass, on dry basis, Max	2.5	—	A-7 of IS 253
vi)	Iron content (as Fe), mg/kg	850 - 1 100	A	—
vii)	Iodine content, mg/kg, Min :			
	a) Manufacturer's level	30	B	—
	b) Distribution channel including retail level	15		
viii)	pH of 5 percent aqueous solution :			
	a) For iron fortification through encapsulated ferrous fumarate	3.5 – 7.5	C	—
	b) For other forms of iron fortification	3.5 – 5.5		
ix)	Sulphate (as SO ₄), percent by mass, Max	1.1	—	A-9 of IS 253
x)	Magnesium (as Mg), water soluble, percent by mass, Max	0.10	—	A-8 of IS 253
xi)	Phosphorous (as P ₂ O ₅), mg/kg :		D	—
	a) For iron fortification through encapsulated ferrous fumarate	Not more than 3 100		
	b) For other forms of iron fortification	2 800 – 3 100		
xii)	Sodium hexa meta phosphate, percent by mass on dry basis, Max	1.0	E	—

AMENDMENT NO. 2 APRIL 2017
TO
IS 16232 : 2014 IRON FORTIFIED IODIZED SALT
(DOUBLE FORTIFIED SALT) — FOOD GRADE —
SPECIFICATION

[Page 1, Table 1, Sl No. x), col (5)] — Substitute ‘A-7’ for ‘A-8’.

(Page 10, Annex F) — Insert the following at the end:

[Page 1, Table 1, Sl No. iv), col (4)] — Substitute ‘G’ for ‘--’.

[Page 1, Table 1, Sl No. iv), col (5)] — Substitute ‘--’ for ‘A-6’.

[Page 1, Table 1, Sl No. v), col (5)] — Substitute ‘A-6’ for ‘A-7’.

[Page 1, Table 1, Sl No. ix), col (5)] — Substitute ‘A-8’ for ‘A-9’.

(Page 7, clause D-2, para 2, line 5) — Insert the following at the beginning:

‘Dry it at $100 \pm 2^\circ\text{C}$ and for 2 h.’

(Page 8, clause E-4.1, para 1, line 1) — Substitute ‘sample’ for ‘sodium hexa meta phosphate’.

(Page 8, clause E-5, para 1, line 1) — Substitute the following for the existing clause:

‘E-5 CALCULATION

$$\begin{array}{l} \text{Sodium hexa meta} \\ \text{phosphate} \\ \text{percent by mass on} \\ \text{dry basis,} \end{array} = \left[60 - \left(\frac{V_1 \times N_1}{N_2} \right) \right] \times \left(\frac{0.06172}{W} \right) \times 100$$

where

V_1 = volume of H_2SO_4 (titre value);

N_1 = normality of H_2SO_4 ;

N_2 = normality of NaOH ; and

W = mass of the material taken for the test, in g.

ANNEX G

[Table 1, SI No. (iv)]

DETERMINATION OF MATTER INSOLUBLE IN DILUTE HYDROCHLORIC ACID

G 1 REAGENTS

G 1.1 Dilute Hydrochloric Acid - 1 : 1 (v/v) and 1 : 20 (v/v).

G 1.2 Concentrated Hydrochloric Acid

G 2 Procedure

Accurately weigh about 20 g of the dried sample, dissolve it in 200 ml of water in a beaker and heat to boiling and cool. Filter the solution through a weighed Gooch crucible G4 or sintered glass crucible and wash the residue till it is free from soluble salts. Dry the crucible along with the insoluble residue to constant mass. Transfer the residue obtained into a beaker. Alternatively, transfer the sintered crucible itself with the residue to 100 ml beaker, and add 15 to 20 ml of water and 25 ml of concentrated hydrochloric acid so that the sinter base is immersed in the acid. Heat to boiling for 10 to 15 minutes to ensure decomposition of the residue with the acid. Remove the sintered crucible from the beaker (if it is transferred to the beaker) and wash the crucible with water. Evaporate the extract and wash solution together to dryness and transfer the solid residue with 25 ml of dilute hydrochloric acid (1: 1).

G.2.1 Filter on a filter paper (Whatman No. 41 or equivalent), wash the precipitate first with warm dilute hydrochloric acid (1: 20) and then with hot water until washings are free from chloride. Dry the residue and ignite in a silica crucible at 550°C for 2 hours. Cool and weigh. NOTE - If silicate drier is not used, acid insoluble residue will be very low for accurate weighing. In that case, repeat with 50 g of dried material.

G.3 Calculation

Acid insoluble matter, percent by mass = $\frac{100 \times M_1}{M_2}$

M_1 = mass in g of the residue, and

M_2 = mass in g of dried sample taken for the test.

(FAD 08)